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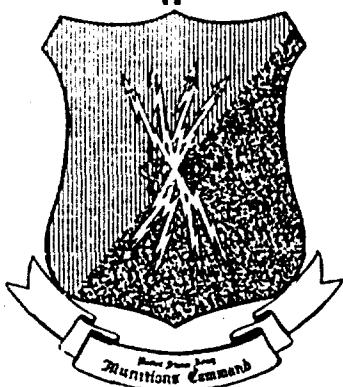
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PICATINNY ARSENAL TECHNICAL REPORT 3040  
LABORATORY STUDIES TO DEVELOP  
AN IMPROVED PROCESS FOR  
THE STABILIZATION OF GUNCOTTON

ANDREW F. SMETANA

JAMES W. McCAHILL

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PICATINNY ARSENAL  
DOVER, NEW JERSEY



**LABORATORY STUDIES TO DEVELOP AN IMPROVED  
PROCESS FOR THE STABILIZATION OF GUNCOTTON**

**BY**

**Andrew F. Smetana  
James W. McCahill**

**June 1963**

**Feltman Research Laboratories  
Picatinny Arsenal  
Dover, New Jersey**

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**Approved by:**

  
**L. H. Eriksen  
Chief, Explosives &  
Propellants Laboratory**

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## ABSTRACT

On the basis of the 100°C Vacuum Stability Test, it is shown that 30 hours of boiling in the presence of a 0.05-0.2 N barium ion solution is at least equivalent to the 70-hour standard hydrolytic boil in distilled water for the stabilization of guncotton. Using radioactive barium-133 and sulfur-35 to trace barium ions and sulfate groups, respectively, a chemical relationship between these two species is observed to exist in the boiling medium. The interpretation is offered from these experiments that the greater part of the total sulfate content in unstabilized guncotton is present as sulfuric acid and the remainder as sulfate half-ester. It is postulated that the action of barium ions or hard water cations in general in accelerating guncotton stabilization involves a mechanism whereby the cations chemically combine with the sulfate impurities in such a manner that these impurities do not harm the fibers.

## RECOMMENDATIONS

It is recommended that an engineering study be conducted to determine the feasibility of substituting 30 hours or less of boiling in the presence of hard water cations for the standard 70-hour hydrolytic boil in the stabilization of guncotton. If this study indicates that such a change is economically advantageous, then a series of pilot plant stabilization runs should be made, using added cations in the water. To eliminate the statistical and experimental variations inherent in the small-scale laboratory experiments described in this report, each pilot plant run should be performed with at least 25 pounds of guncotton.

## INTRODUCTION

Previous work at Picatinny was concerned with the development of techniques for determining the sulfate impurity in stabilized cellulose nitrate (Ref 1) and the investigation of different methods for removing this impurity (Ref 2) from freshly nitrated fibers. The results obtained from successive aqueous and non-aqueous precipitations of unstabilized cellulose nitrate (11.8 - 13.4% nitrogen) from solutions in various organic solvents gave the tentative conclusion that approximately 90% of the sulfate content of water-washed product is readily removable sulfuric acid; the remainder, which is more tenaciously held in the fibers, may be a true sulfate ester. Dilute acid boil treatment of cellulose nitrate for up to sixty hours does not eliminate all of the readily removable acid and leaves the more firmly bound sulfate virtually unchanged.

Another phase of this project involved a study of the removal of occluded acids from freshly nitrated cellulose by cold saline leaching (Ref 3). This approach was based on investigations by Sheppard that were first reported in 1921 (Ref 4). It was observed that extraction with salt solution causes a greater release of acidity than extraction with distilled water. Of the total acidity released, the bulk is sulfuric acid and the remainder is nitric acid. The amount of acidity released by saline leaching is inversely related to the nitrogen content of the NC, that is, less acid is extracted when the NC has a higher nitrogen content.

An evaluation was made of the use of a cold saline leach in combination with short aqueous boils to produce stable cellulose nitrate. Samples were subjected to both the 100° C Vacuum Stability Test and the 134.5° C German Heat Test. The 100° C Vacuum Stability Test was found to be superior in detecting the differences between samples. The vacuum stability test showed that a combination of a 4-hour cold saline leach and a 10-hour water boil, used prior to the standard heating and poaching operation, resulted in pyrocellulose (12.6% N) comparable in stability to samples boiled in water for 40 hours. However, the substitution of a saline leach for boiling time appeared to have no advantage for guncotton (13.4% N).

The potential value of the cold saline leach for pyrocellulose, however, was judged to be limited, since in recent years the belief has become widespread among cellulose nitrate chemists that the aqueous boiling time for this product can be reduced from fifty hours to twenty hours without any adverse effect on the stability of the final material. Reduction of the stabilization time is currently of interest primarily in connection with high-grade cellulose nitrate. Since this study showed that saline leaches are least effective for higher nitrogen content cellulose nitrates, the use of this treatment for stabilization does not offer much advantage. The consideration of cold saline leaches as a stabilization procedure was therefore abandoned. This report describes the results of some small-scale laboratory experiments on the stabilization of guncotton by the direct inclusion of salts derived from hard cations in the boiling medium. This approach was based on observations by Forest Products Laboratory (Ref 5) that the stabilization of cellulose nitrate is accelerated by the use of commercial processing water (i.e., hard as compared with distilled water). The objective of the approach taken at Picatinny Arsenal was to find a possible relationship between the cation content of process water and the ease of stabilization, and the mechanism involved.

## DISCUSSION

The presence of calcium in commercial processing waters would normally dictate the use of this cation for the stabilization experiments. However, the use of a radioactive form of a hard water cation in later experiments was also planned, to study the retention of such an ion during stabilization. Although barium is not usually present in commercial hard waters, this ion was selected for the bulk of the investigation because of the excellent nuclear properties of barium-133 as a radiotracer (half-life 7.2 years, gamma energy 0.36 Mev). For this particular purpose, it was believed that the chemistry of barium would be similar to that of calcium.

The standard stabilization treatment (Ref 6) for guncotton consists of a 70-hour hydrolytic boil with fresh water changes after 60 and 65 hours. This is followed by a mechanical beating operation and a poaching treatment made up of one 4-hour, one 2-hour, and two 1-hour hydrolytic boils.

The first phase of this study consisted of a series of guncotton stabilization experiments in which barium and calcium acetate, in various concentrations and with boiling periods of 20 and 30 hours, were substituted for the standard 70-hour hydrolytic boil. As a basis for comparison, experiments were also conducted using 30- and 70-hour boils with distilled water as a medium. Each treatment was then followed by the standard beating and poaching operation and the dried sample of guncotton was then subjected to the 100° C Vacuum Stability Test (Ref 7).

The maximum readable result of this test, as performed at Picatinny Arsenal, is 11 ml of gas in a 40-hour period. Values in excess of this amount are recorded as 11+ ml, with the time to reach the value indicated. For purposes of this discussion, no arbitrary quantity of gas will be designated as an indication of acceptable stability but the numbers will be used for comparison purposes only as a measure of relative thermal stability. The results of these experiments are given in Table 1.

In treatment with barium salt in the range between 0.05 N and 0.2 N, the concentration of the solution does not appear to be important. However, the boiling period between 20 and 30 hours does seem to have a critical effect on stabilizing ability. Of 9 samples treated with barium acetate solution for 20 hours, eight gave 11+ ml of gas in 40 hours or less whereas only one gave a readable result of 5.65 ml. On the other hand, for the 30-hour treatment with barium acetate solution, 8 out of 10 samples tested gave readable results with an average of 5.14 ml of gas, and 2 gave 11+ values.

Because only a very few runs were made with calcium solution, the results are not as conclusive as those obtained in the barium experiments, but here again the relative advantage of 30 hours of boiling over 20 hours is indicated.

With distilled water boiling, all of 9 samples treated for 30 hours gave 11+ values in 40 hours or less. Of 7 samples boiled for 70 hours, 6 gave readable values averaging 5.62 ml of gas whereas 1 yielded 11+ ml. On the basis of the 100°C Vacuum Stability Test, a 30-hour hydrolytic boil in the presence of barium ions in concentrations between 0.05 and 0.2 N is at least equivalent in stabilization ability to a 70-hour distilled water boil. The advantage of added cations to the boiling medium is thus shown.

TABLE 1  
Guncotton stabilization experiments

Sample No.	% Nitrogen	Boiling Time, hrs	100°C Vacuum Stability, ml of gas/40 hr
<b>0.05 N BARIUM ACETATE BOILING TREATMENT</b>			
3-61	13.51	20	11+ in 18 hrs
4-61	13.54	20	11+ in 40 hrs
7-61	13.62	20	11+ in 19 hrs
3-61	13.51	30	7.82
4-61	13.54	30	9.36
7-61	13.53	30	7.08
<b>0.1 N BARIUM ACETATE BOILING TREATMENT</b>			
1-61	13.51	20	5.65
2-61	13.48	20	11+ in 40 hrs
5-61	13.46	20	11+ in 40 hrs
6-61	13.48	20	11+ in 40 hrs
1-61	13.46	30	0.26
2-61	13.45	30	11+ in 40 hrs
5-61	13.46	30	5.74
6-61	13.49	30	9.53
JM-17	13.44	30	0.23

TABLE 1 (Continued)

Guncotton stabilization experiments

Sample No.	% Nitrogen	Boiling Time, hrs	100° C Vacuum Stability, ml of gas/40 hr
<b>0.2 N BARIUM ACETATE BOILING TREATMENT</b>			
6-61	13.53	20	11+ in 24 hrs
7-61	13.57	20	11+ in 40 hrs
6-61	13.53	30	11+ in 40 hrs
7-61	13.43	30	1.08
<b>0.05 N CALCIUM ACETATE BOILING TREATMENT</b>			
3-61	13.58	20	11+ in 18 hrs
3-61	13.58	30	11+ in 40 hrs
<b>0.1 N CALCIUM ACETATE BOILING TREATMENT</b>			
1-61	13.56	20	11+ in 40 hrs
2-61	13.55	20	11+ in 40 hrs
1-61	13.56	30	8.35
2-61	13.56	30	11+ in 40 hrs
JM-16	13.51	30	0.13
<b>DISTILLED WATER BOILING TREATMENT</b>			
1-61	13.56	30	11+ in 16 hrs
2-61	13.56	30	11+ in 16 hrs
3-61	13.56	30	11+ in 18 hrs
4-61	13.60	30	11+ in 16 hrs
5-61	13.61	30	11+ in 16 hrs
6-61	13.73	30	11+ in 16 hrs
7-61	13.66	30	11+ in 20 hrs
JM-14	13.57	30	11+ in 20 hrs
JM-18	13.40	30	11+ in 40 hrs
1-61	13.56	70	10.33

TABLE 1 (Continued)

Guncotton stabilization experiments

Sample No.	% Nitrogen	Boiling Time, hr	100°C Vacuum Stability, ml of gas/40 hr
2-61	13.57	70	7.49
3-61	13.57	70	11+ in 40 hrs
4-61	13.64	70	1.62
5-61	13.61	70	2.81
6-61	13.72	70	9.93
7-61	13.66	70	1.37

As another phase of this study, it was of interest to obtain information about the mechanism by which hard water cations in the boiling medium cause shortened stabilization time for guncotton. As stated previously, instability in cellulose nitrate can be attributed primarily to sulfate or sulfuric acid residues in the fibers. It was therefore desired to observe whether sulfate and cation concentration are in any way related in the hydrolytic boiling process. If one assumes a purely chemical relationship between sulfate and cation, Table 2 shows the theoretically possible relationships that may exist between barium ions and the sulfate impurities which have been postulated as being present in unstabilized cellulose nitrate.

TABLE 2

## Possible relationship between barium ions and sulfate impurities

Sulfate Impurity	Product Resulting from Chemical Interaction with Ba <sup>++</sup> ions	Molar Ratio Ba/SO <sub>4</sub>
Occluded sulfuric acid	BaSO <sub>4</sub>	1
Sulfate half-ester	(ROSO <sub>3</sub> ) <sub>2</sub> Ba	0.5
Sulfate full-ester	No reaction	---

From previous work in this field (Ref 2), it was known that the sulfate content in freshly nitrated unstabilized guncotton is about 1% by weight and that, when boiled in distilled water, the sulfate is exponentially

removed with time. Preliminary experiments with barium solution boiling showed that here the sulfate becomes essentially fixed in the fibers after approximately 20 to 30 hours. It is postulated that, in distilled water, occluded sulfuric acid is leached out of the fibers whereas, in a barium solution, at least a portion of the sulfate becomes chemically or physically fixed in the fibers. Therefore, to facilitate interpretation of residual barium-to-sulfate molar ratios, a series of experiments was conducted in which freshly nitrated guncotton was boiled in various combinations of distilled water and 0.1 N barium acetate solution treatments. To facilitate chemical analysis, the sulfate was tagged with sulfur-35 by using tagged sulfuric acid in the original nitration mixed acid, and the barium acetate was tagged with Ba-133 in the boiling medium. By measuring the radioactivity of each nuclide in a sample after treatment, quantitative analyses for both sulfate and barium were obtained in concentration levels that would be extremely difficult to analyze by more conventional techniques.

The results of these experiments, with corresponding residual barium and sulfate contents, in percent and calculated molar ratios of each are shown in Table 3. All experiments were conducted in duplicate to show reproducibility. In the first four experiments, the distilled water pre-boil was varied from 0 to 30 hours while the barium acetate final treatment was held constant at 30 hours. Under these conditions, residual molar ratios of barium to sulfate decreased from greater than one to 0.6 -0.7 as the initial boiling period in distilled water is increased. In the last three experiments, the distilled water pre-boil was varied from 10 to 20 to 30 hours, followed by barium solution treatments of 20, 10, and 4 hours, respectively. Here it is seen that, as the initial distilled water boiling time is increased, with a corresponding decrease in barium solution treatment, the residual molar ratio of barium to sulfate is decreased from above 0.5 to below 0.5. The following interpretation is offered to account for the results of these experiments.

In freshly nitrated unstabilized guncotton, the greater part of the residual sulfate content in the fibers is in the form of occluded sulfuric acid. When boiled for 30 hours in the presence of barium ions, preceded by only a short distilled water boil or none, the sulfate becomes fixed or precipitated in the fibers as insoluble barium sulfate with a resulting  $Ba : SO_4$  ratio of close to 1 (Experiments 1 and 2). As the period of distilled water pre-boil is increased to 30 hours, most of the occluded sulfuric acid is released and subsequent boiling with barium ion solution results in the formation of the barium salt of sulfate half-ester with a corresponding molar ratio approaching 0.5 (Experiments 3 and 4). Values

TABLE 3

## Retention of barium and sulfate in guncotton

Sample No.	Duration of Distilled Water Pretreatment, hr	0.1 N Barium Acetate	% Ba	% SO <sub>4</sub>	Molar Ratio Ba : SO <sub>4</sub>
1-62	0	30	0.75	0.42	1.2
16-61	0	30	0.70	0.38	1.3
1-62	10	30	0.30	0.24	0.9
14-61	10	30	0.22	0.16	1.0
1-62	20	30	0.19	0.19	0.7
14-61	20	30	0.11	0.12	0.6
1-62	30	30	0.15	0.15	0.7
14-61	30	30	0.10	0.10	0.7
1-62	10	20	0.26	0.24	0.8
14-61	10	20	0.19	0.19	0.7
1-62	20	10	0.09	0.18	0.3
14-61	20	10	0.10	0.15	0.5
1-62	30	4	0.05	0.15	0.2
14-61	30	4	0.06	0.13	0.3

below 0.5 for molar ratios were encountered in Experiments 6 and 7 because the time was insufficient for barium ion interaction with the sulfate half-ester.

Admittedly, the actual results in these experiments do not support the above interpretation in a clear-cut manner. For example, it is difficult to explain a molar ratio of greater than 1 in Experiment 1 except to conjecture that the sample, after treatment, also contains some occluded barium acetate or barium combined in some way other than with sulfate. In Experiments 3 and 4, molar ratios of 0.5 were not quite achieved. Here is is conjectured that the residual sulfate is still composed of some occluded sulfuric acid. Nevertheless, the interpretation offered appears reasonable in light of the data, at least in a qualitative sense. The final values for sulfate content, as shown in Table 3, also substantiate the

above interpretation to some degree. In the guncotton samples from Experiments 3 and 4, where essentially sulfate half-esters are postulated as the residues, these sulfate contents are in the order of 0.10% to 0.19%. Assuming an initial content of 1%, these values represent between 10% and 20% of the total initial sulfate content present in unstabilized guncotton as chemically bound sulfate esters. This is in agreement with what had previously been suggested using a different experimental approach (Ref 2).

The mechanism for guncotton stabilization can thus be postulated as involving the removal of sulfate acid impurities from the fibers or otherwise rendering them innocuous. In a cation-free or low-salt-concentration medium, a long boil is needed to free the occluded acid residues and also to hydrolyze the smaller quantities of chemically bound sulfate esters. The final heating and poaching operation serves to open the fibers even more to drive out the more tenaciously held or bound impurities. Added cations such as barium or hard water ions do not accelerate the release of these sulfate impurities but combine with them chemically in such a manner that they can no longer harm the fibers.

## EXPERIMENTAL PROCEDURE

### Nitration of Cotton to Guncotton

The appropriate quantities of nitration grade cotton linters were teased, dried at 100°C for two hours, and weighed. From 40 g to 50 g of linters was used for each nitration. The mixed acids listed in Table 4 were used for the preparation of the indicated batches of cellulose nitrate. In each nitration, an amount of mixed acid equal to 40 times the weight of cotton linters was used. When cellulose nitrate samples were to be analyzed for sulfate content, the mixed acids were tagged with sulfuric acid containing sulfur-35. The cotton linters and mixed acid were left in contact, with occasional mixing, for 24 minutes at 34-36°C.

The mixture was then transferred to a large Buchner funnel and pressed as dry as possible with the aid of suction and a plastic dowel. The cellulose nitrate was then transferred in several portions with vigorous stirring to a 4-liter beaker containing distilled ice water, and was allowed to soak with occasional stirring for 20 minutes. The cellulose nitrate was washed with distilled water on a Buchner funnel over Whatman No. 1 filter paper until the pH of the wash water became constant (pH 4.0-4.5) and was then separated into the number of portions required for the intended stabilization experiment.

TABLE 4

Composition of mixed acids used for preparation of cellulose nitrate samples

Sample No.	Sulfuric Acid, %	Nitric Acid, %	Water, %
1-61	63.69	29.64	6.67
2-61	63.69	29.64	6.67
3-61	64.26	28.01	7.43
4-61	64.26	28.01	7.43
5-61	65.28	27.23	7.49
6-61	64.99	27.46	7.55
7-61	64.92	27.08	8.00
14-61	64.64	27.41	8.05
16-61	64.64	27.41	8.05
1-62	64.78	27.38	7.85
JM-14	64.18	28.43	7.39
JM-16	64.81	27.67	7.52
JM-17	64.81	27.67	7.52
JM-18	64.81	27.67	7.52

Treatment of Untagged Guncotton

The distilled-water-washed cellulose nitrate batches were treated as follows: Portions of each batch were boiled in distilled water for 30 and 70 hours and for 20 and 30 hours in 0.05 N, 0.1 N, or 0.2 N barium or calcium acetate solutions.

During the 70-hour distilled water boiling treatment, the distilled water was drained and fresh water was added after periods of 60 and 65 hours. On completion of the boiling treatment, the samples were rinsed several times with distilled water and placed in a Valley-type beater with 400 ml of distilled water. The beater was operated for 60 seconds with the blades at the more separated position and for 30 seconds at the closer position.

The slurry was filtered on a Buchner funnel and subjected to the poaching treatment, which consisted of a 4-hour, a 2-hour, and two 1-hour boiling treatments each in fresh solutions of the same composition as was used in the previous boiling treatment.

The cellulose nitrate samples were then rinsed on a Buchner funnel with five 400-ml portions of distilled water, air dried overnight, and dried under vacuum at 50-55°C for 6 hours.

The dried samples were then subjected to the 100°C Vacuum Stability Test which is described in Picatinny Arsenal Technical Report 1401, Revision 1.

#### Treatment of Sulfur-35 Tagged Guncotton

Freshly prepared, distilled-water-rinsed cellulose nitrate tagged with sulfur-35 was separated into the number of portions needed for the experiment.

Samples to be analyzed for sulfate were treated with non-radioactive 0.1 N barium acetate, and those to be analyzed for barium were treated with 0.1 N barium acetate tagged with barium-133. In each of the following cases involving 0.1 N barium acetate treatment, two portions were treated, one for the analysis of sulfate content and the second for the analysis of barium content. Two portions of cellulose nitrate were boiled with 0.1 N barium acetate for 30 hours. A portion was boiled for 10 hours with distilled water and separated into four subportions, two to be boiled for 20 and two for 30 hours with 0.1 N barium acetate solutions.

One portion was boiled for 20 hours with distilled water and separated into four subportions, two to be treated for 10 hours and two for 30 hours with 0.1 N barium acetate solutions.

The last portion was boiled for 30 hours with distilled water and separated into four subportions, two to be boiled for four hours and two for 30 hours with the 0.1 N barium acetate solutions.

Two liters of fresh solution were used for each boiling treatment. Each individual sample was rinsed with five 400-ml portions of distilled water, air dried overnight, and dried under vacuum at 50-55°C for six hours. The dried samples were analyzed for barium and sulfate contents.

#### Analysis of Guncotton Samples for Sulfate Content

A Packard Tri-Carb Liquid Scintillation Counter (Model 314) was used for the radioanalysis of sulfur content of treated guncotton samples. The settings used on the instrument included a high voltage of 1140 volts, with pulses recorded from 10 volts to infinity.

The scintillator gel used for the counting medium was composed of 15 g of Thixcin and 4.00 g of PPO (2,5 diphenyl-oxazole) in one liter of CP toluene. This mixture was homogenized in a Waring Blender for three minutes (Ref 8).

Sulfur-35 tagged guncotton samples were counted in the Tri-Carb instrument. These counts were related to a reference standard previously assayed for sulfate content by the method of Hoffpauir & Guthrie as modified by Castorina et al (Ref 2). The reference was counted in the same manner as the experimental samples to obtain a relation between counting rate and sulfate content.

Approximately 100 mg of guncotton was weighed out to the nearest tenth of a milligram in a tared 5-dram glass counting vial supplied by Wheaton Glass Co. To this vial was added 21 ml of scintillator gel mixture. The vial was capped, shaken vigorously in order to disperse the guncotton as uniformly as possible in the scintillation medium, and counted. Three determinations were made on each sample. Background count determinations were made on vials containing portions of scintillation gel.

#### Analysis of Guncotton for Barium Content

A Baird-Atomic Gamma Spectrometer was used to measure the radioactivity of barium-133 in the guncotton. All pulses from 0.25 Mev to infinity were recorded. Barium sulfate prepared by precipitation from the Barium-133 tagged 0.1 N barium acetate solution was used as a reference standard for the determination of barium content in the barium-133 tagged guncotton samples. The applicability of this procedure was proved by comparing the count rate for samples of guncotton containing barium-133 before and after oxidation of the samples by a mixture of nitric and sulfuric acids.

Guncotton (100-200 mg) and standard samples were placed for count rate determination in one-dram glass vials supplied by the Kimble Glass Company. A background count rate determination was run on the glass vials.

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<p>Accession No. <u>AD</u> Picatinny Arsenal, Dover, N. J.</p> <p><b>LABORATORY STUDIES TO DEVELOP AN IMPROVED PROCESS FOR THE STABILIZATION OF GUN-COTTON</b></p> <p>Andrew F. Smetana, James W. McCahill</p> <p>Technical Report 3040, June 1963, 15 pp., tables. OMS 5610.11.70300, DA Proj 5B99-04-011. Unclassified report</p>	<p>1. Cellulose nitrates — Stabilization</p> <p>I. Smetana, Andrew F. II. McCahill, James W.</p> <p>UNITERMS</p> <p>Stabilization Guncotton Boiling Barium acetate Smetana, A. F. McCaill, J. W.</p> <p>On the basis of the 100°C Vacuum Stability Test, it is shown that 30 hours of boiling in the presence of a 0.50-0.2 N barium ion solution is at least equivalent to the 70-hour standard hydrolytic boil in distilled water for the stabilization of guncotton. Using radioactive barium-133 and sulfur-35 to trace barium ions and sulfate groups, respectively, a chemical relationship</p>
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